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IS 8167 (1989): Method for determination of reducibility index of iron ore oxides, lumps oore, sinter and pillets [MTD 13: Ores and Raw Materials]

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IS 8167 : 1989

Indian Standard

METHOD FOR DETERMINATION OF REDUCIBILITY INDEX OF IRON OXIDES, LUMP ORE, SINTER AND PELLETS

(*First Revision*)

UDC 622 341·1 : 543·82

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1990 Price Group 3

Ores and Raw Materials Sectional Committee, MTD 13

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 25 August 1989, after the draft finalized by the Ores and Raw Materials Sectional Committee had been approved by the Metallurgical Engineering Division Council

This standard was first published in 1976 In this revision, the scope of standard has been extended to iron ore pellets also and the method of testing has been aligned with the corresponding International Standard

The reducibility indicates a measure of the ease with which oxygen combined with iron could be removed from iron ore lump, sinter and pellets, with a reducing gas at the time of reduction and gives one of the important characteristics of the lump ore, sinter and pellets which is given due consideration in its utilization This standard has been prepared to establish uniform practice for the determination of reducibility of iron ore lump, sinter and pellets.

This standard differs from IS 11292 : 1985 'Method of determination of relative reducibility of iron oxides' lump ores, sinter and pellets' in that it determines the reducibility index, the rate of reduction, *dR/dt* at 40 percent reduction, without specifying a definite period of reduction.

In the preparation of this standard, assistance has been derived from ISO 4695 : 1984 'Iron ores — determination of reducibility', issued by the International Organization for Standardizatian (ISO)

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

AMENDMENT NO. 1 NOVEMBER 1993
TO
IS 8167 : 1989 METHOD FOR DETERMINATION OF REDUCIBILITY INDEX OF IRON OXIDES, LUMP ORE, SINTER AND PELLETS

(First Revision)

(*Page* 2, *clause* **7.3.2**, *line* 2) — Substitute '50' *for* '15'.

(*Page* 2, *clause* **9.3**) — Replace the existing matter with the following:

'The reduction tube shall be made of heat-resistant non-scaling metal to withstand temperatures of higher than 950°C. The perforated plate is mounted in the reduction tube for supporting the test portion. The height of the support should be so adjusted that the tube is in the constant temperature zone of the furnace. A typical illustration of reduction tube is shown in Fig 3 given on next page.'

(*Page* 2, *clause* **9.5**, *line* 2) — Substitute '1' *for* '5'

(*Page* 3, *clause* **10.6**, *line* 2) — Substitute 'four' *for* 'three'

Ores and Raw Materials Sectional Committee, MTD 13

FOREWORD

This Indian Standard (First R
1989, after the draft finalized
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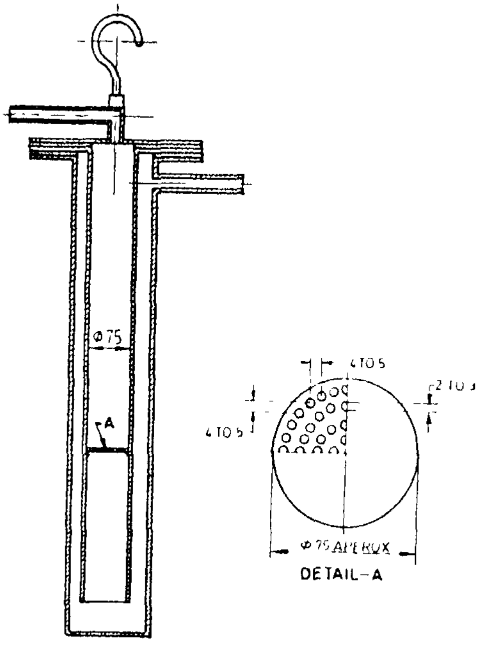


Thickness of plate 4 mm.

FIG. 3 TYPICAL EXAMPLE OF REDUCTION TUBE

(MTD 13)

Reprography Unit, BIS, New Delhi, India

Indian Standard

METHOD FOR DETERMINATION OF REDUCIBILITY INDEX OF IRON OXIDES, LUMP ORE, SINTER AND PELLETS

(First Revision)

1 SCOPE

1.1 This standard prescribes the method for the determination of reducibility index of iron oxides; lump ore, sinter and pellets under specific conditions.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard

IS No.	*Title*
460 (Part 1) : 1985	Specification for wire cloth test sieves (*third revision*)
1493 : 1969	Methods of chemical analysis of iron ores
1493 (Part 1) . 1981	Methods for determination of common constituents

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 Percentage Reducibility

The ratio of oxygen removed during the test to the total available oxygen for reduction present in the sample, expressed as a percentage.

3.2 Reducibility Index

Rate of reduction, percentage loss of oxygen per minute, at 40 percent reducibility of the sample and designated as dR/dt at 40 percent reduction

4 PRINCIPAL OF TEST

4.1 The reduction sample is placed into a reduction tube which is inserted into a furnace and connected to a weighing device The tube is heated to the specified temperature while inert gas is passing through it The reducing gas is then admitted, replacing the inert gas During the reduction the sample is weighed continuously and the loss in mass recorded at specified time intervals. At the end of the test the sample is cooled in a stream of purified inert gas.

5 NUMBER OF TESTS

5.1 The reduction test shall be carried out in duplicate. The period of reduction of the second test shall be exactly as that of the first test, as given in **10.5**.

6 SAMPLES

6.1 The test sample shall be prepared from the sample obtained for physical tests A quantity of sample of approximately 2 kg sized according to **6.2**, shall be prepared (500 g each, sufficient for two tests and 1 kg for chemical analysis)

6.2 The size range of the test sample for ores, sinters or pellets shall be in the ranges of − 12·5 mm to + 10 mm. Alternate size ranges may also be used by agreement between the parties concerned However, particles with size greater than 16 mm shall not be tested, using the apparatus described. Test sample of ore or sinter shall be prepared by sieving to remove all materials passing through 10 mm sieve, and crushing carefully all the material over 12·5 mm to just pass through 12·5 mm sieve After crushing, any material or size under 10 mm generated during the process, shall be removed by sieving through 10 mm sieve Test sample of pellets in the size range 10 mm to 12·5 mm shall be obtained by sieving, pellets of size above 12·5 mm and those below 10 mm shall be rejected.

6.3 The test sample shall be oven dried at 105 ± 5°C to remove the moisture contents and cooled in a desccicator before subjecting to tests

6.4 Mass of each reducibility test sample should be approximately 500 g. A 1 000 g test portion shall be used for chemical analysis, for the determination of total iron and ferrous iron, so that the amount of oxygen combined with iron may be calculated These constituents shall be determined by the methods specified in IS 1493 1969 and IS 1493 (Part 1) : 1981.

7 REDUCING GAS

7.1 The reducing gas should consist of 30 ± 1·0 *v/v* percent carbon monoxide (CO) and 70 ± 1·0 *v/v* percent nitrogen (N_2 and should be passed through drying towers or U-tubes containing blue silica gel in duplication following the removal of CO_2, O_2 and other impurities.

7.2 The maximum allowable impurities in the reducing gas should be 0·5 percent hydrogen (H_2) and 0·8 percent carbon dioxide (CO_2).

7.3 Temperature and flow rate of reducing gas shall be as given in **7.3.1** and **7.3.2**.

7.3.1 The reducing gas before entering the basket, holding the sample should be preheated to 950 ± 10°C. This preheating should be attained in the same reactor.

7.3.2 The reduction gas flow should, during the entire test period, be maintained at 15 litres per minute at standard temperature and pressure.

8 TEMPERATURE OF TEST

8.1 During the entire test period, the temperature of the reduction sample should be maintained at 950 ± 10°C throughout the entire bed of the sample.

9 APPARATUS

9.1 The apparatus shall consists of the following:

a) A leak proof system to supply and regulate the flow of nitrogen and carbon monoxide gases.

b) A reduction tube.

c) A continuous weighing device to determine the loss of mass of the sample at any time during the test.

d) An electrically heated furnace to heat the entire test portion to the specified temperature. and

e) A gas burner or a device to eliminate the unused carbon monoxide emerging from the exit end of the reduction tube.

9.2 The reduction vessel consists of a tube with suitable top and bottom to make it leakproof. The heating zone should be so adjusted that the lower part of the zone may be utilized for heating the reducing gas to desired temperature. The reduction tube is suspended in an electrically heated furnace, from a balance which allows indication of the variation of the mass of the sample continuously during reduction. Nitrogen and reduction gas taken from pressure gas cylinders, or other sources, are expanded, dried and introduced into the reduction tube. The waste gas is drawn off from the upper part of the tube. Caution should be exercised to dispose off the waste gas by burning or by some other appropriate means. A regularly calibrated thermocouple, suitably positioned, should be used to record the temperature. A typical arrangement of apparatus is shown in Fig. 1.

9.3 The reduction tube shall be made up of heat-resisting non-scaling steel. The height of the support should be so adjusted that the tube is in the constant temperature zone of the furnace. A typical illustration of reduction tube with basket is shown in Fig. 1.

9.4 The furnace shall be capable of attaining a temperature of the sample to 950 ± 10°C in 60 minutes. The entire test sample shall be maintained at this temperature throughout the test period.

9.5 Weighing Device

The sensitivity of the weighing device should be ± 5 g and with a suitable load capacity. The sensitivity should be checked regularly. The initial mass of the test sample (m_0) shall be recorded with the sensitivity.

NOTE — A load capacity of 20 kg will be generally suitable for this test.

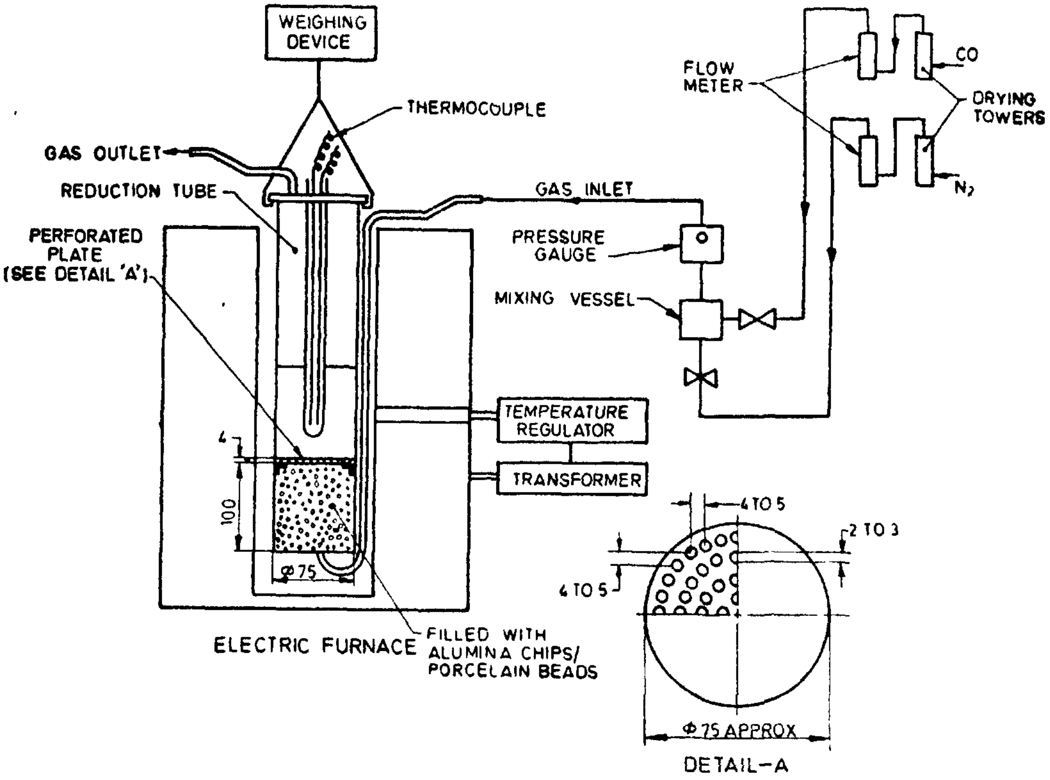
9.6 Sieves

Sieves having square mesh apertures of 10 mm and 12·5 mm shall be used, conforming to IS 460 (Part 1) : 1985.

10 PROCEDURE

10.1 The test sample of approximately 500 g, shall be placed in the reduction tube and shall be evened out by tapping. In order to have a uniform gas flow, a two layer bed of chips or beads of size 10 to 12·5 mm diameter of heat exchange medium shall be placed between the perforated plate and the test sample as shown in Fig. 1. The tube holding the sample shall then be introduced into the reduction tube and attached to the balance so that it suspended freely inside the furnance without any contacts with its walls.

10.2 The air in the tube is replaced with nitrogen gas. The sample is heated and while heating, a flow rate of approximately 5 litres per minute at NTP of inert gas is passed through the sample. As the temperature of the sample approaches 950°C, the flow rate of the nitrogen is increased to 15 litres per minute at NTP.



All dimensions in millimetres.

FIG. 1 SCHEMATIC DIAGRAM OF REDUCTION APPARATUS

10.3 The sample shall then be allowed to soak at this temperature for 30 minutes minimum until the mass is constant. The mass of the sample, immediately before reduction, shall be weighed and recorded as m_1. The reduction gas shall then be introduced to replace nitrogen.

10.4 The product gases coming out of the reduction tube shall be burnt before letting out to the atmosphere.

10.5 The mass of sample should be recorded every 3 minutes initially for first 15 minutes and thereafter 10 minutes interval.

10.6 The test shall be performed until reducibility of 60 percent is reached. If after three hours this has not been attained, the test shall be terminated. The mass of the sample at the end of test shall be recorded as m_2. The sample shall than be cooled under a flow of purified nitrogen.

11 REPORT OF RESULTS

11.1 Calculation of Reducibility

The reducibility is calculated from the experimental data and the chemical composition of the sample according to the equation:

$$R = \frac{m_1 - m_2}{m_0\,(\,0.42\,W_1 - 0.111\,W_2\,)} \times 10^4$$

where

R = reducibility, percent, at the end of the test;

m_0 = initial mass of the test portion, in g;

m_1 = mass of the test sample, immediately before starting the reduction, in g,

m_2 = mass of the test sample at the end of reduction in g;

W_1 = total iron content (% Fe) of the test sample prior to the test, in percent; and

W_2 = ferrous oxide content (% FeO) of the test sample prior to the test, in percent.

11.2 Calculation of *dR* at 40 Percent Reduction

It shall be calculated from the following formula, rounded to two places of decimals:

$$\frac{dR}{dt} = \frac{33{\cdot}6}{(t_{60} - t_{30})}$$

where

t_{60} = time in minutes to attain 60 percent reducibility (Rt_{60}) obtain as per **11.3**.

t_{30} = time in minutes to attain 30 percent reducibility (Rt_{30}) obtained as per **11.3**.

NOTE — In case 60 percent reducibility is not attained during the test, lower values may be accommodated by using the formula given below:

$$\frac{dR}{dt} = \frac{K}{(t_y - t_{30})}$$

where

t = time in minutes to attain y percent reduction obtain as per **11.3**.

If Y = 50 percent then k = 20·4.

If Y = 55 percent then k = 26·4.

11.3 Calculation of the reduction time, at which a desired reducibility is attained, by the sample.

11.3.1 From the data available in **10.6**, graphs shall be plotted, if points, indicating loss of sample mass in grams against reduction time in minutes, scaled linearly, covering the entire test period, for each of the two tests, carried out as per **11.4**. The points shall be joined by a smooth curve.

11.3.2 The loss of mass of the sample, corresponding to the desired reducibility R_1 (as required under **11.2**) shall be calculated from the following equation:

$$\text{Loss of sample mass (g)} = R_1 \times m_0 (0{\cdot}43\, W_1 - 0{\cdot}111\, W_2) \times 10^{-4}$$

where

R_1 = desired reducibility (percent), and

m_0 = W_1 and W_2 are as described in **11.1**,

11.3.3 From the loss of mass of the sample, calculated as per **11.3.2**, the corresponding reduction time shall be read from the curve.

11.3.4 The value of *dR*/*dt* at 40 percent reduction, shall be calculated from values obtained in **11.3.3**, accoording to **11.2**, and reported as per **11.4.1**.

An example of graphical presentation of the curve indicating loss of sample mass/reduction time, relation and calculation of *dR*/*dt* values, is given Andex A.

11.4 Expression of Results

11.4.1 *Reducibility Index*

dR/*dt* at 40 percent reduction, shall be reported, as the arithmetic mean of the reduction time required to attain 30 percent reduction and 60 percent reduction (or 50 percent or 55 percent reduction, as the case may be, as per **11.2**) read from both the curves, of the pair of tests, rounded to second decimal places.

11.4.2 *Reducibility*

Values shall be reported as the arithmetic mean of both the reducibility values, calculated for the pair of tests, and reported to the first decimal place.

12 TEST REPORT

12.1 The report shall include the following:

a) Description of the sample,

b) Total iron (%) and FeO (%) of the sample before reduction,

c) *dR*/*dt* at 40% reduction,

d) Reducibility,

e) Loss of mass/reduction time curves, and

f) Reference to this standard.

ANNEX A

(*Clause* 11.3.4)

GRAPHICAL PRESENTATION INDICATING LOSS OF MASS/REDUCTION TIME RELATION AND CALCULATION OF *dR/dt* VALUES

A-1 An example of graphical presentation of the curves indicating of a sample of iron ore pellets is given in Fig. 2.

A-2 A typical example of the calculation of *dR/dt* at 40 percent reduction from Fig. 2 is given below:

Total Fe	(W_1)	64·67%
FeO	(W_2)	0·14%
Sample mass (m_0) Test 1		500·00 g
Sample mass (m_0) Test 2		500·00 g

Loss of sample mass

$= m_0 (0{\cdot}43\ W_1 - 0{\cdot}111\ W_2)\ 10^{-4} \times R_1$

(for both tests)

$= 500 (0{\cdot}43 \times 64{\cdot}67 - 0{\cdot}111 \times 0{\cdot}14) 10^{-4} \times R_1$

NOTE — m_0 for both the tests is identical in this case.

R_1	*Loss of Sample Mass* (1·389 6 × R_1)	*Corresponding Reduction Time as Read from the Curve* Fig. 1 (minutes)		*Arithmetic Mean of Reduction Time of the Two Tests*
(%)	(s)	Test 1	Test 2	
(a)	(b)	(c)	(d)	(c) + (d)/2
60	83·38	124·0	118·0	121·00
30	41·69	39·0	35·5	37·25

dR/dt at 40 percent reduction

$$= \frac{33{\cdot}6}{t_{60} - t_{30}}$$

$$= \frac{33{\cdot}6}{121 - 37{\cdot}25}$$

$$= 0{\cdot}40$$

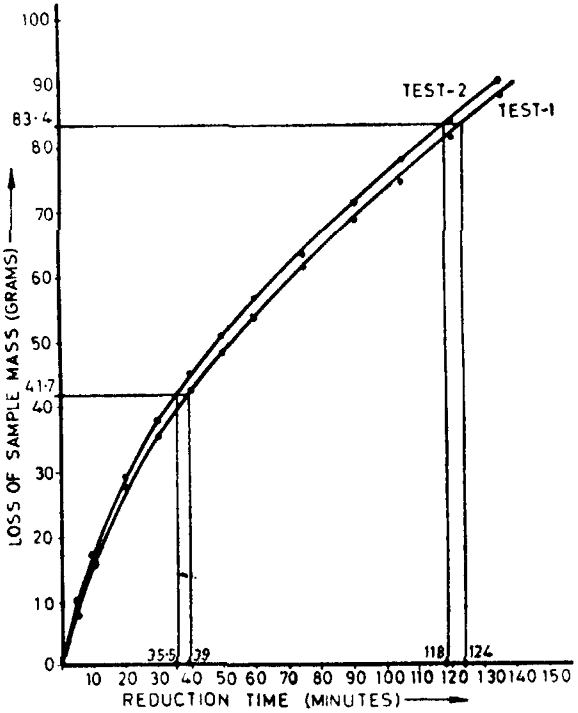


FIG. 2 LOSS OF MASS/REDUCTION TIME RELATION CURVE

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